

Table 2. Bond lengths (Å), angles (°) and selected torsion angles (°) with *e.s.d.*'s in parentheses

C1—O1	1.383 (5)	C5—C6	1.385 (6)
C1—C2	1.396 (6)	C6—C7 ⁱ	1.527 (6)
C1—C6	1.395 (6)	C8—C9	1.484 (13)
C2—C3	1.392 (6)	C8—C10	1.554 (10)
C2—C7	1.520 (6)	C1G—C2G	1.40 (4)
C3—C4	1.383 (6)	C1G—C7G	1.53 (2)
C4—C5	1.385 (6)	C2G—C3G	1.41 (6)
C4—C8	1.526 (7)	C3G—C4G	1.40 (6)
O1—C1—C2	119.7 (3)	C1—C6—C7 ⁱⁱ	121.2 (4)
O1—C1—C6	118.7 (4)	C5—C6—C7 ⁱⁱ	120.5 (4)
C2—C1—C6	121.6 (4)	C2—C7—C6 ⁱⁱⁱ	112.7 (4)
C1—C2—C3	117.7 (4)	C4—C8—C9	112.6 (6)
C1—C2—C7	121.7 (4)	C4—C8—C10	111.1 (5)
C3—C2—C7	120.6 (4)	C9—C8—C10	113.2 (6)
C2—C3—C4	122.2 (4)	C2G—C1G—C7G	120 (2)
C3—C4—C5	118.3 (4)	C1G—C2G—C3G	120 (3)
C3—C4—C8	120.4 (4)	C2G—C3G—C4G	120 (4)
C5—C4—C8	121.3 (4)	C2G—C1G—C2G ⁱ	120 (2)
C4—C5—C6	121.9 (4)	C3G—C4G—C3G ⁱ	120 (3)
C1—C6—C5	118.3 (4)		
C1—C2—C7—C6 ⁱⁱⁱ	−88.8 (5)	C3—C4—C8—C9	−117.5 (6)
C1—C6—C7 ⁱⁱ —C2 ⁱⁱ	88.4 (5)	C3—C4—C8—C10	114.3 (5)

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} - y, z$; (ii) $\frac{1}{2} - y, x, z$; (iii) $y, \frac{1}{2} - x, z$.

Programs used: *SHELXS86* (Sheldrick, 1985); *SHELX76* (Sheldrick, 1976); *ORTEPII* (Johnson, 1976); *The Universal Crystallographic Computing System—Osaka* (1979).

The occupancy of the guest molecule was refined as variable; the final refined occupancy showed the molecular ratio of host to guest as 1:1. The H atom of the OH group has two orientations.

There are two circular frameworks including four O—H...O hydrogen bonds as clockwise and anticlockwise rotations. The positive peaks of these H atoms were indistinct on the difference Fourier maps.

There are not enough strong interactions to hold the guest molecule fixed rigidly although several weak interactions are observed. This causes the high *R* value and the high temperature factors of the guest molecule.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55703 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1013]

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.

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Structure of MoAs₂O₇: corrigendum and addendum. By E. HUMS, *Siemens AG, Bereich Energieerzeugung (KWU), Hammerbacherstrasse 12 und 14, D - 8520 Erlangen, Germany* and H. BURZLAFF and W. ROTHAMMEL, *Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstrasse 10, D-8520 Erlangen, Germany*

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Abstract

Data collection and refinement for the title compound, molybdenum diarsenate, were repeated; the results are presented.

The crystal structure of the title compound was solved and published by the authors (Hums, Burzloff & Rothammel, 1991). At that time the refinement did not allow the application of anisotropic displacement parameters for all

atoms, since one $\beta(2,2)$ parameter ran negative. Moreover, unusually high residua could be recognized in the difference Fourier map. Therefore the data collection was repeated using more stable conditions for the measuring temperature and extending the observed region of the reciprocal space such that averaging for symmetrically equivalent reflections was possible. The sample and the instrument were not changed. The new data allowed a complete refinement as usual; the *Experimental* contains the crystal data and the experimental procedure relating to the new data collection. Table 1 contains the structural

parameters.* The strongest extremes in the difference map are located close to the Mo and As atoms.

The crystal structure consists of layers oriented parallel to (101) built by centrosymmetric Mo₂O₁₀ groups. The double octahedra are connected to chains along [010] by As atoms with tetrahedral O-atom coordination. These chains are connected by the other As atoms with coordination number three (trigonal pyramid). This type of As atom provides a connection between the layers with shortest As—O distance of 2.89 Å. Fig. 1 shows a complete projection of the structure; Fig. 2 shows the vibrational ellipsoids for all atoms. Distances and angles do not show significant changes; all deviations are less than 0.03 Å and 0.7°.

Experimental

Crystal data

As₂MoO₇

$M_r = 358$

Monoclinic

$P2_1/n$

$a = 16.14 (1) \text{ \AA}$

$b = 4.550 (3) \text{ \AA}$

$c = 7.788 (5) \text{ \AA}$

$\beta = 100.07 (1)^\circ$

$V = 563 \text{ \AA}^3$

$Z = 4$

Mo $K\alpha_1$ radiation

$\lambda = 0.70926 \text{ \AA}$

Cell parameters from 51 reflections

$\theta = 6.5\text{--}12.7^\circ$

$\mu = 13.901 \text{ mm}^{-1}$

$T = 293.0 (1) \text{ K}$

Needle

$0.32 \times 0.048 \times 0.008 \text{ mm}$

Honey coloured

Data collection

Modified PW 1100 diffractometer

$\omega/2\theta$ scans

Absorption correction: analytical

$T_{\min} = 0.546$, $T_{\max} = 0.909$

2237 measured reflections

888 independent reflections

888 observed reflections

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 24.0^\circ$

$h = -18 \rightarrow 18$

$k = -5 \rightarrow 5$

$l = -8 \rightarrow 1$

4 standard reflections

frequency: 120 min

intensity variation: 0.99–1.03%

Refinement

Refinement on F

Final $R = 0.033$

$wR = 0.021$

$S = 2.0$

888 reflections

92 parameters

$w = 1/\sigma(F)$

$(\Delta/\sigma)_{\text{max}} < 0.01$

$\Delta\rho_{\text{max}} = 1.24 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -1.13 \text{ e \AA}^{-3}$

Extinction correction: Larson (1970)

Extinction coefficient: 483 (38)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Program(s) used to solve structure: SIR88 (Burla *et al.*, 1989).

All other computations performed with a version of CRYSTAN88 (ATARI, 1989) adapted to a PC 386.

* Lists of anisotropic displacement parameters, distances and angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55606 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1012]

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

	x	y	z	U_{eq}
Mo	0.09911 (4)	0.0005 (1)	0.15991 (8)	0.0097
As1	0.05763 (4)	0.4606 (1)	0.82515 (9)	0.0092
As2	0.21193 (4)	0.5757 (1)	0.6156 (1)	0.0118
O1	0.1260 (3)	0.2582 (9)	0.9669 (6)	0.0121
O2	0.1051 (3)	0.587 (1)	0.6647 (6)	0.0141
O3	0.1251 (3)	0.220 (1)	0.3338 (6)	0.0168
O4	0.0252 (3)	0.7602 (9)	0.9266 (6)	0.0092
O5	0.9755 (3)	0.260 (1)	0.7284 (6)	0.0127
O6	0.1837 (3)	0.784 (1)	0.1638 (7)	0.0183
O7	0.2369 (3)	0.207 (1)	0.6758 (6)	0.0180

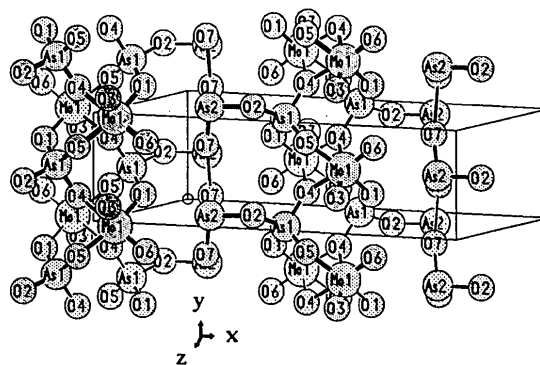


Fig. 1. Projection of one layer in (101) passing the end of the base vectors a and c .

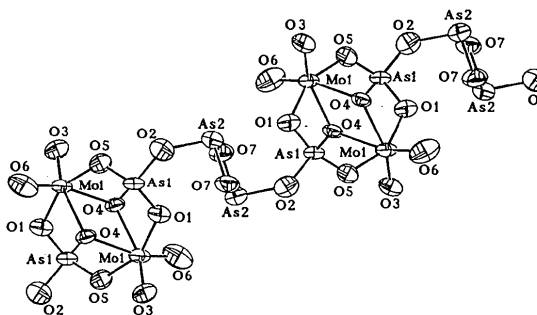


Fig. 2. Displacement ellipsoids (99% probability); projection of the same part of the structure as in Fig. 1 along b .

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